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Metallurgical Analysis of Wear Particles and Wearing Surfaces

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Metallurgy Division
Institute for Materials Research
National Bureau of Standards
Washington, D. C. 20234

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Quarterly Report
Contract 4–8049

Prepared for
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U. S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary

NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

I. OBJECTIVE

The objective of the project has been to characterize the wear particles and surface degradation produced by wear in bearing and gear tests in which the effect of several variables on failure of the wearing surfaces has been examined. The information obtained has been correlated with the results of allied studies conducted by others in an attempt to develop an understanding of the processes producing wear and degradation of metal surfaces in sliding, rubbing, rolling, and/or rotating contact and the effects of lubricants, lubricant additives, bearing materials, etc. on these processes. The characterization of the wear particles and wearing surfaces should aid in the establishment of the interrelationships between wear particle shape, size, size distribution, chemical compositions, metallurgical structure, and surface damage prior to failure.

II. APPROACH

The initial approach has been concerned primarily with the examination of particles recovered by the Ferrographic technique from samples of lubricating oils taken periodically during tests and service of bearings, gears, sliding surfaces, etc., in which such experimental variables as lubricants, lubricant additives, bearing materials, loads, etc., have been studied. Our examinations have been conducted principally using both Scanning and Transmission Electron Microscopy techniques, observing particle shapes, sizes, surface structures and other parameters as functions of distance along the Ferrogram and determining a semi-quantitative elemental chemical analysis of selected and typical particles. These electron microscope techniques have been used to characterize the wear particles and associated surface degradation produced in the bearing and gear tests conducted by others. They provide information on particles and surface details too small for study by optical microscopy methods.

After suitable techniques have been developed for examining and characterizing both the wear particles and the surfaces that produced them, these methods will be used to examine and evaluate specimens removed or obtained from programmed wear tests being conducted on associated projects.

III. RESULTS

Particle Microanalysis

Correct quantitative identification of particles and determination of their composition in the size range below 100 μm requires careful experimental measurements and the performance of periodic calibrations in order to insure accuracy. The particles of interest here may have been worn from various components of the system under study or may be foreign impurities drawn into that system. X-ray emission from bulk materials under electron beam excitation is an analytical process that has been studied in detail in recent years. However, X-ray emission from particulates has not been sufficiently well examined. At the electron beam voltages of interest (about 10-30kV) considerable electron penetration of thin particles occurs and significantly affects the accuracy of X-ray analysis.

Calculations have been conducted using the Monte Carlo technique of the X-ray emission from spheres and cylinders of nickel and iron having diameters below 100 μm. The technique calculates electron trajectories through the solid and provides information on the generation of X-rays. Figure 1 shows the calculated trajectories in copper for three different electron energies. The increasing penetration with increasing energy is indicated. The calculations provide the required corrections relative to X-ray emission from bulk materials. They would apply to particle analysis as conducted in this program. Figure 2 shows the calculated values for iron cylinders of various diameters up to 10 μm. The intensity ratio relative to a bulk sample is given. The curve also applies fairly closely to the case of spherical particles. A particle 1 μm in size is predicted to generate only about 50% of the X-ray intensity of bulk material. Particles larger than 2 μm can be analyzed without significant thickness correction required. Experimental measurements have been made on nickel wire and on particles filed from NBS-SRM 483, an Fe-3 Si alloy, certified for bulk chemical homogeniety.

A specimen of the particulates mounted on a Be substrate is shown in Figure 3. An individual particle is also shown having a tip section of diameter smaller than 1 μm. The experimental results given in Figure 2

are in satisfactory agreement with the calculated curve. The measured intensities fall about 10% too low in the size range 2 to 10 μm. This may be due to the rough surface topography of the particles and to difficulties in dimensional measurements. Data are also available for aluminum and for nickel particles.

Ferrograph Evaluation

Experiments have been conducted to explore the effect of oil sample dilution on subsequent Ferrograph analysis. The aim here was to determine the quantitative capability for sizing particles through the Ferrograph method. A Ferrogram was obtained of an oil sample from an RM8B engine. The particles were then removed as completely as possible from the Ferrogram, introduced into fresh oil at a dilution of 5:1 relative to the original sample, and reevaluated. The ratio of initial deposit volume and the percent area coverage ratios at 54 mm and 50 mm are given in Table I. These results indicate that quantitative comparisons can be made under these conditions to an accuracy of about 30%.

A second experiment designed to indicate the quantitative capability of the Ferrograph using particles of controlled size and magnetic moment has been conducted. Porous silica spheres of diameter about 5 μm have been obtained and impregnated with nickel to serve as ferromagnetic particles of known size, shape, and magnetic moment. Initial experiments have involved dilutions of the 5 μm spheres in SAE 30 oil at two concentrations having a ratio of 10:1. Ferrograms were produced from those two samples. Figure 4 shows one of the Ferrograms as seen in the optical microscope.

It is clear that the particle deposit density decreases with distance down the Ferrogram as expected. The individual strings of particles are seen to generally be well formed in this case where particle shape is constant and particle size lies within fairly narrow limits. Figure 5 shows the 5 μm spheres as observed in the SEM (not a Ferrogram specimen). X-ray measurements of the nickel content of randomly selected spheres were conducted. The results are shown in Figure 5, giving the time required to accumulate 5,000 counts under controlled conditions. That

data indicate some nonuniformity in Ni content of the spheres and appear to be related to the optical appearance of some of the spheres. Table I gives the results of this dilution experiment. The initial deposit volume ratio was 11 for the 10:1 dilution, a satisfactory agreement. It is believed that two measures provided by the Ferrogram may be reasonably quantitative indicators of the particle distributions in size and in different oil samples. These are (1) initial deposit volume and (2) optical density at the 50 to 54 mm locations.

Preliminary experiments were conducted with a mixture of nickel-coated spheres of size 5 μm and 0.7 μm. That specimen will permit an evaluation of size selection capability. Optical photographs of two regions on one Ferrogram are shown in Figure 6. Those experiments will continue.

Examination of Bench and Field Test Specimens

Wear particle analyses of oil specimens have continued in conjunction with evaluation of Ferrograms at Trans-Sonics. F2925 was produced from an oil sample from bearing #126 tested at SKF. That oil sample had been stored for some period of months and changes appeared to have taken place relative to earlier Ferrogram examinations. In addition various particle types were analyzed in order to verify certain optical microscope identification procedures. Figure 7 shows the initial deposit region, including a large nonmagnetic particle. Figure 8 shows this same region at higher magnification. The agglomeration and contamination of particles is seen in the right side of this area as compared to the left side region where usual wear flakes are seen. Figure 9 depicts two areas on this Ferrogram where the strings contain oxide particles, clumps and one spheroid. An extraneous brass particle is shown in Figure 10.

Another Ferrogram involving oil from a jet engine that experienced bearing failure was examined. It was found to contain numerous spheroidal particles of unusual appearance and transparency. An example of these spheroids is shown in Figure 11. Analysis of several spheroids was conducted and the results are shown in Figure 12. In all cases these rough, porous spheroids contain much less iron that the lamellar wear particles. Their mode of formation and significance with regard to the failure or wear processes is under study.

Figure Captions

Fig. 1. Monte Carlo calculation results for copper showing electron trajectories and X-ray emission.

Fig. 2. Reduction in X-ray emission vs. particle size.

Fig. 3. Particles of SRM 483 (Fe - 3Si) produced by filing (a) on Be substrate, (b) submicron tip.

Fig. 4. Ferrogram of nickel-coated silica spheres, 5 μm diameter, 2000:1 dilution. F2897.

Fig. 5. Nickel-coated SiO_2 spheres and X-ray emission results.

Fig. 6. Ferrogram of nickel-coated silica spheres, mixture of 5 μm and 0.5 μm diameter, 2000:1 dilution. F2902.

Fig. 7. F2925. Ball bearing test, SKF #126, 293 M revs, entrance deposit. Large particle is transparent, blue, contains Al, Si, small Fe.

Fig. 8. F2925. Area of entrance deposit.

Fig. 9. F2925. Strings of wear particles. (a) ide particle (dark) is iron oxide with sulfur present. (b) Spheroid is Fe, as is end clump.

Fig. 10. F2925. Extraneous brass particle associated with iron particle string.

Fig. 11. F2916. Jet engine oil, bearing failure. Rough speroidal particles.

Fig. 12. F2916. X-ray analysis of (a) spheroid and (b) iron wear particles. Full scale 5×10^3 cts.

Table I. FERROGRAPH EVALUATION - DILUTION EFFECTS

Sample	Initial Deposit		Percent Area Coverage			
	Volume	Height	Initial	54 mm	50 mm	10 mm
K9	6.6×10^6	65 µm	36	14	5	4
1/5:K9*	1.0×10^6	24 µm	11	2.2	0.7	1
ratio	6.6	---	--	6.3	7.1	4
NBS-7	1.2×10^6	23 µm	14	5	0.6	0.5
1/10:NBS-7	0.11×10^6	12 µm	2.5	0.6	---	---
ratio	11	---	--	8.3	---	---

*Particles were washed from original Ferrogram

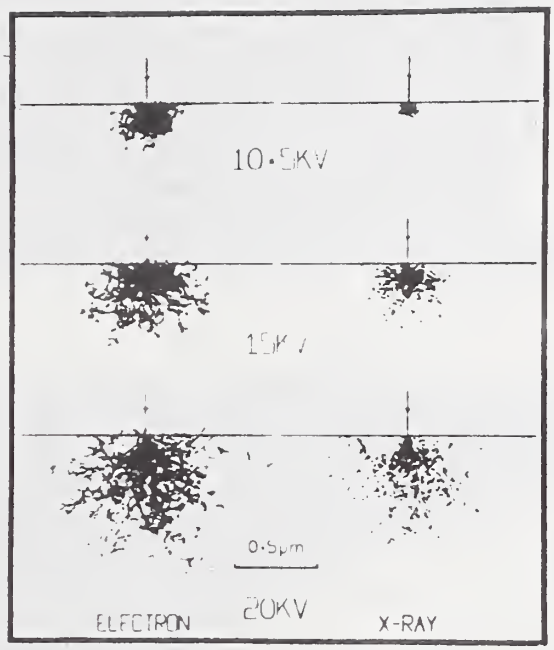


Fig. 1 Monte Carlo calculation results for copper showing electron trajectories and X-ray emission.

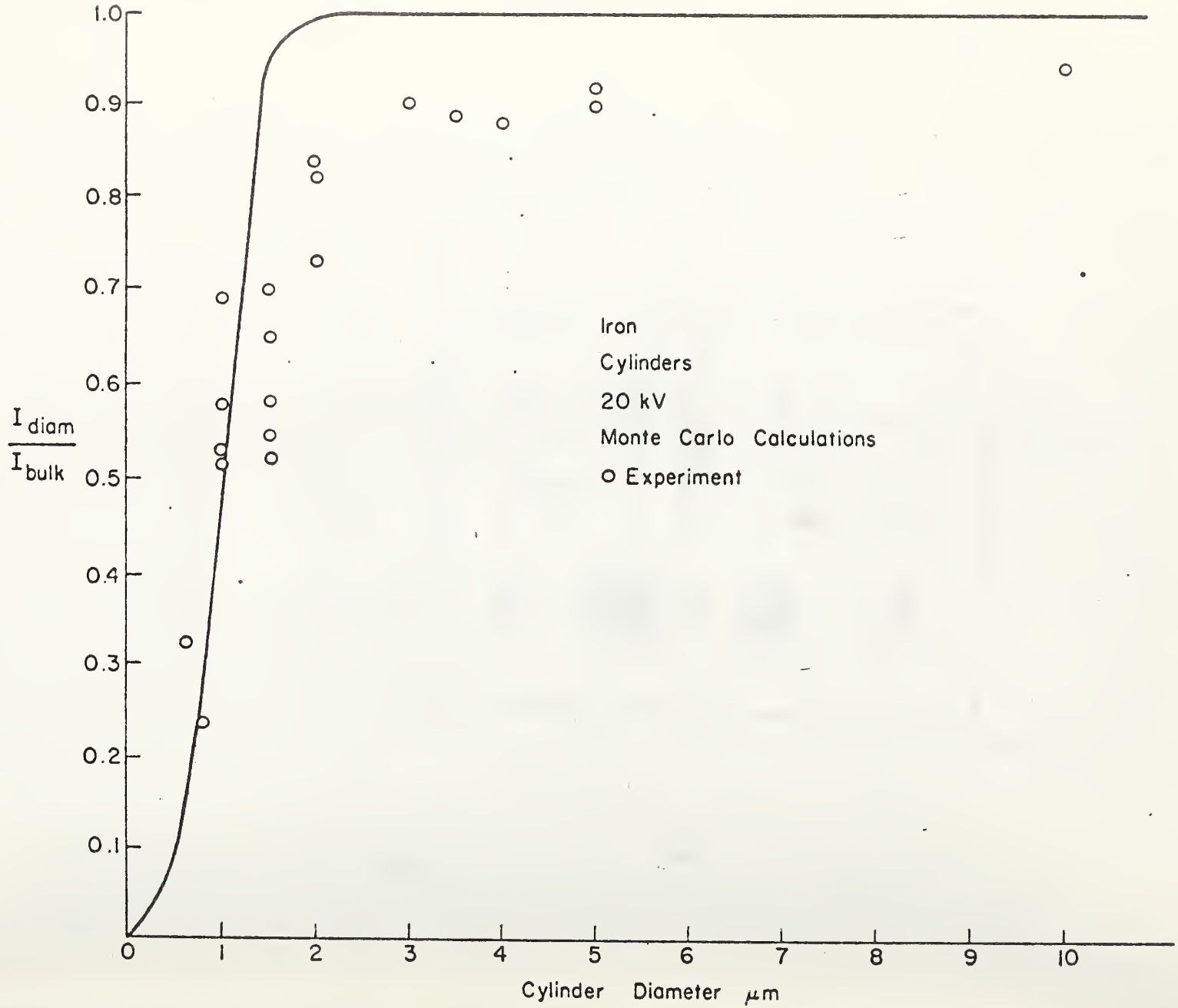


Fig. 2 Reduction in X-ray emission vs. particle size

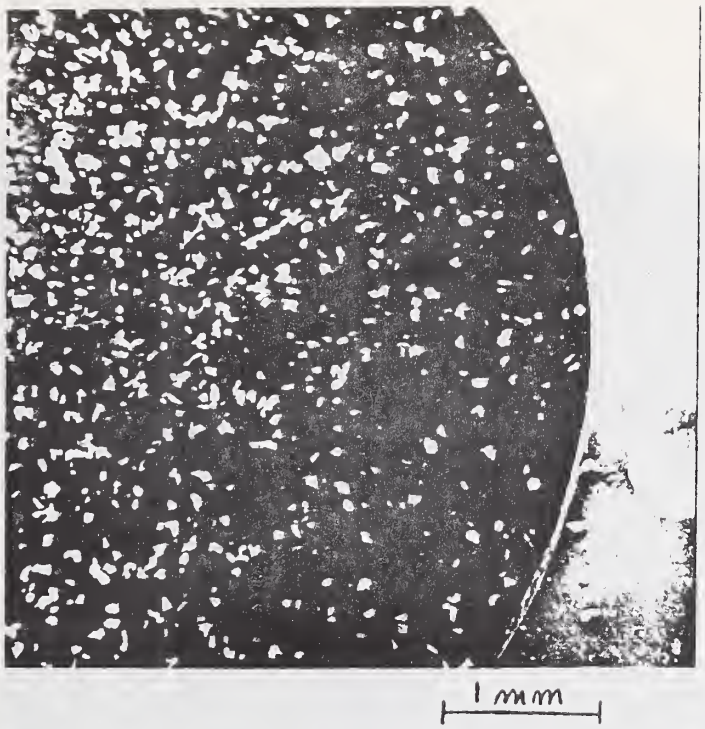


Fig. 3 Particles of SRM 483 (Fe - 3Si) produced by filing (a) on Be substrate, (b) submicron tip.

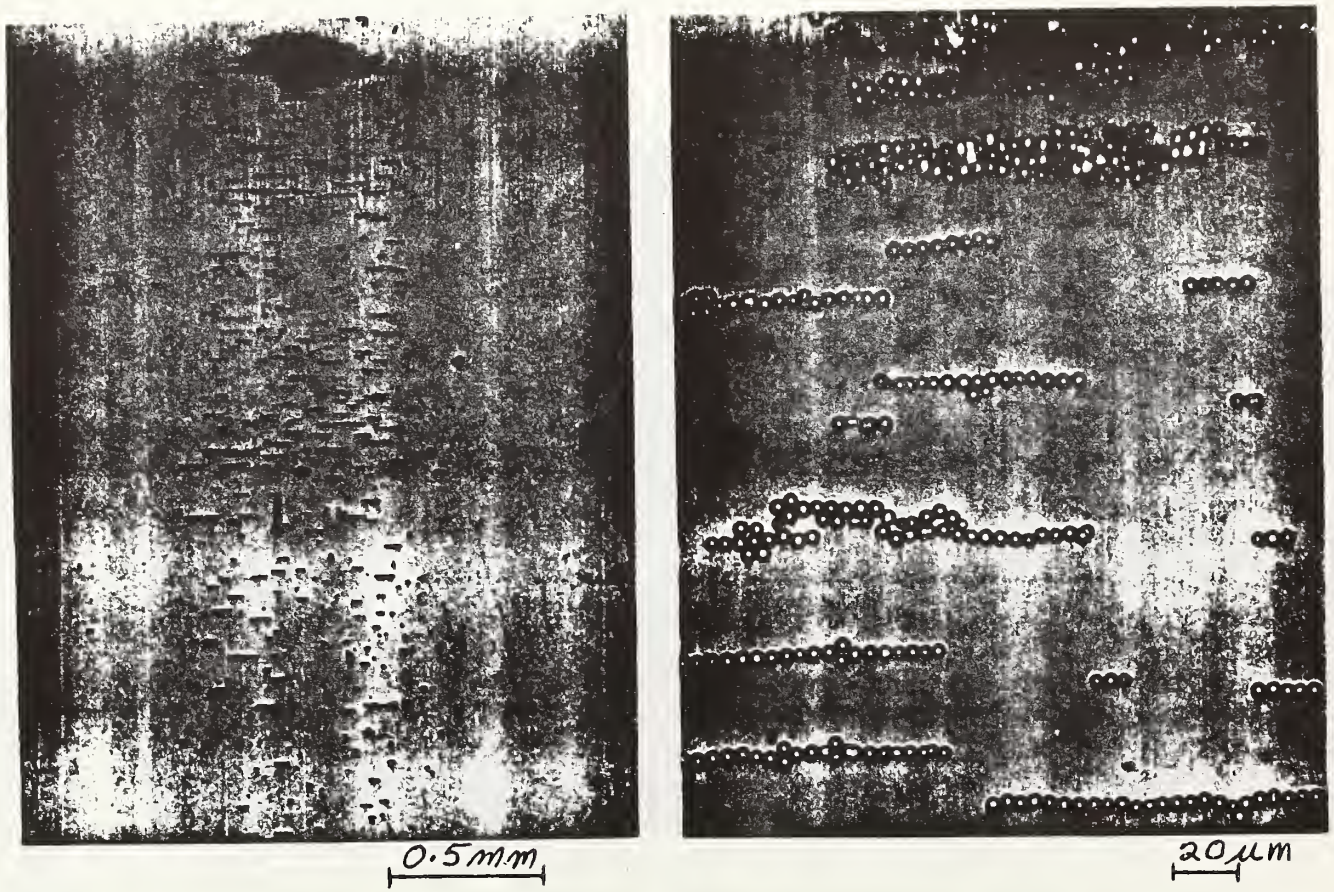
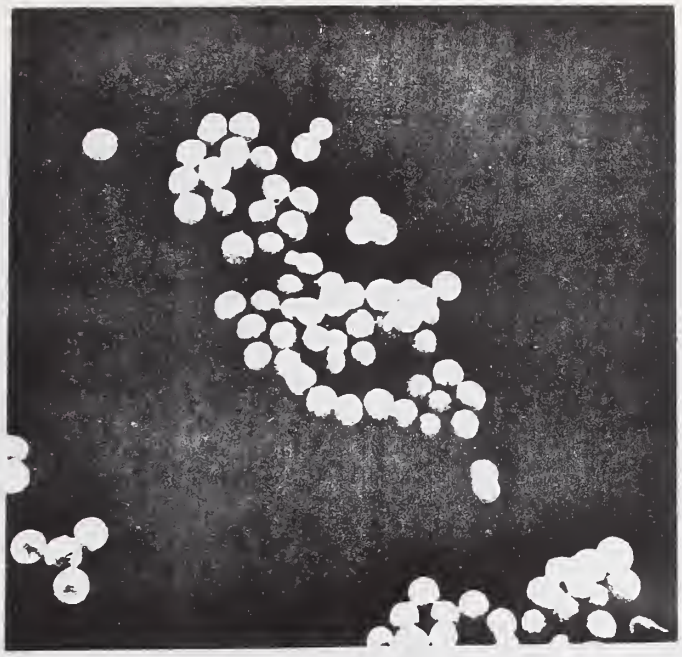
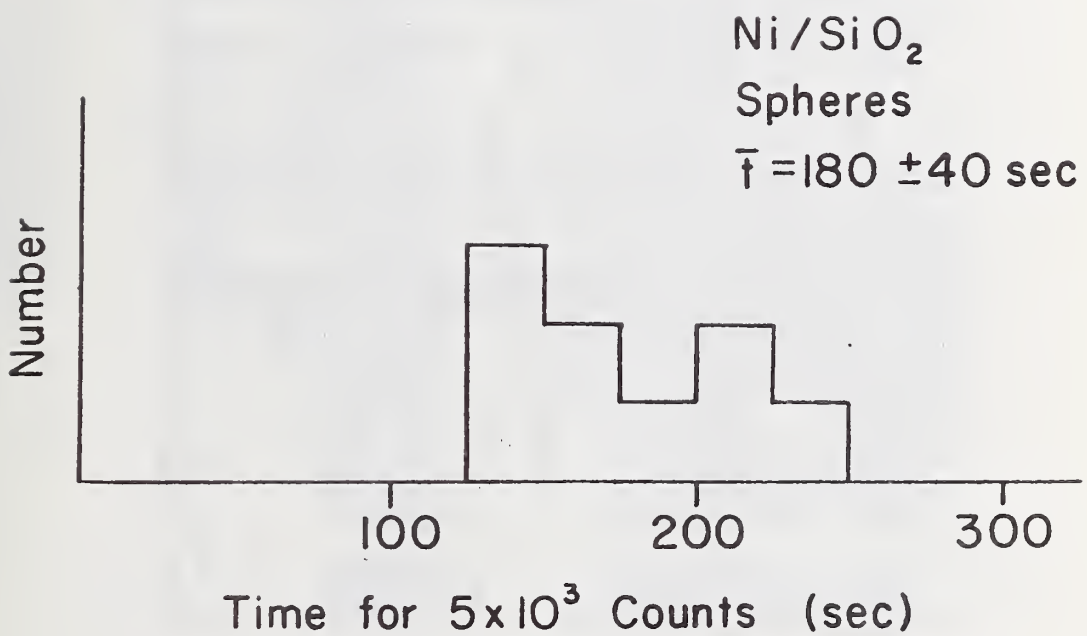
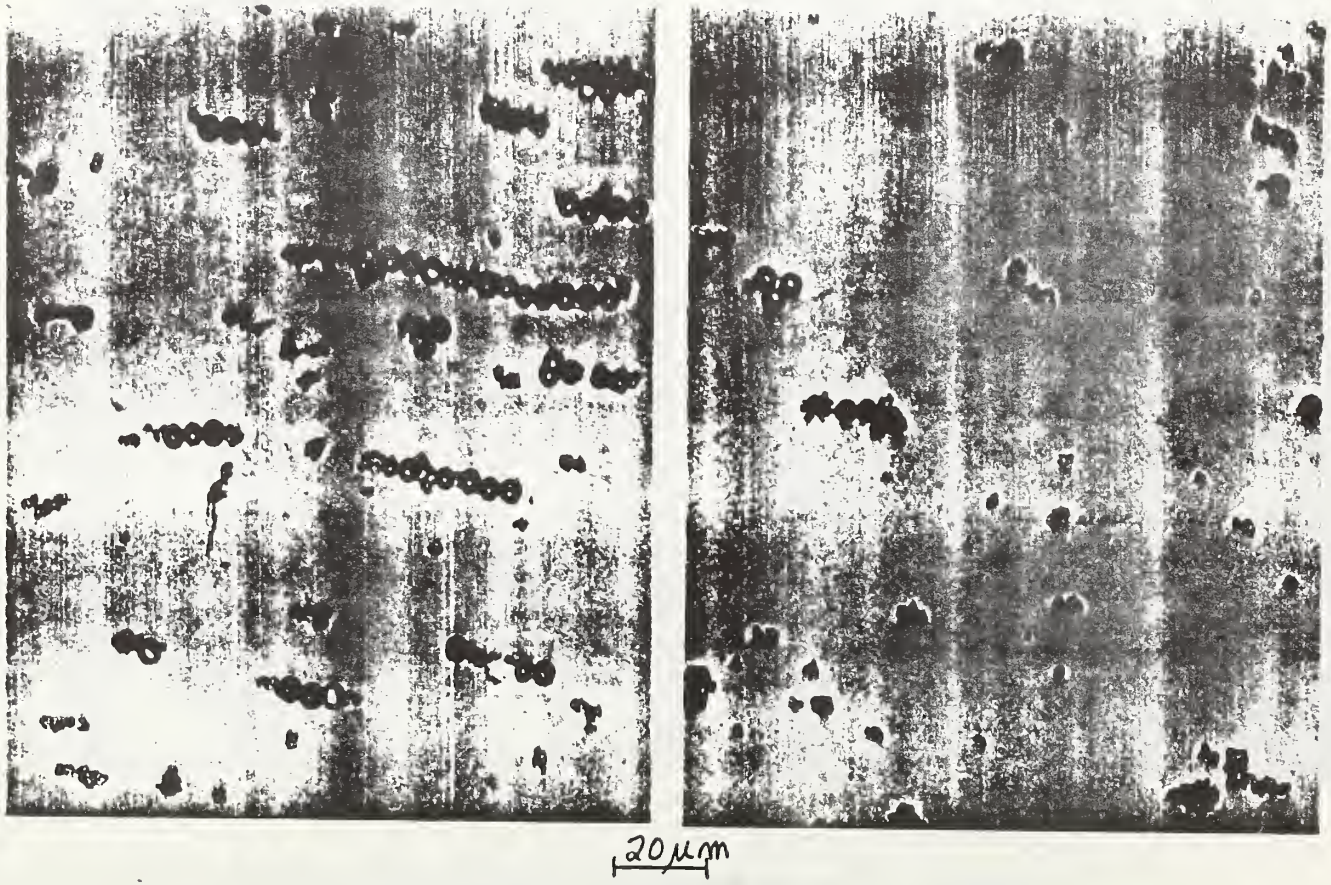


Fig. 4 Ferrogram of nickel-coated silica spheres, 5 μm diameter, 2000:1 dilution. F2897.

Fig. 5 Nickel-coated SiO_2 spheres and X-ray emission results.







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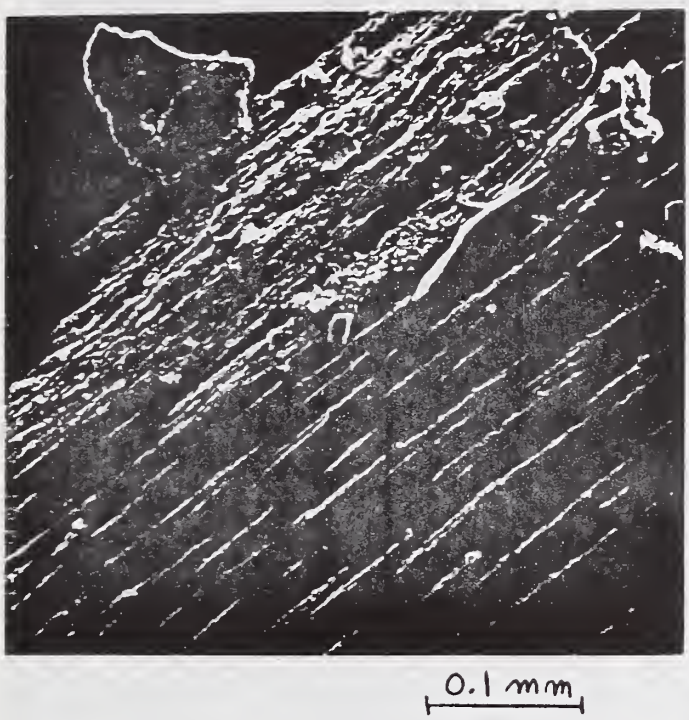

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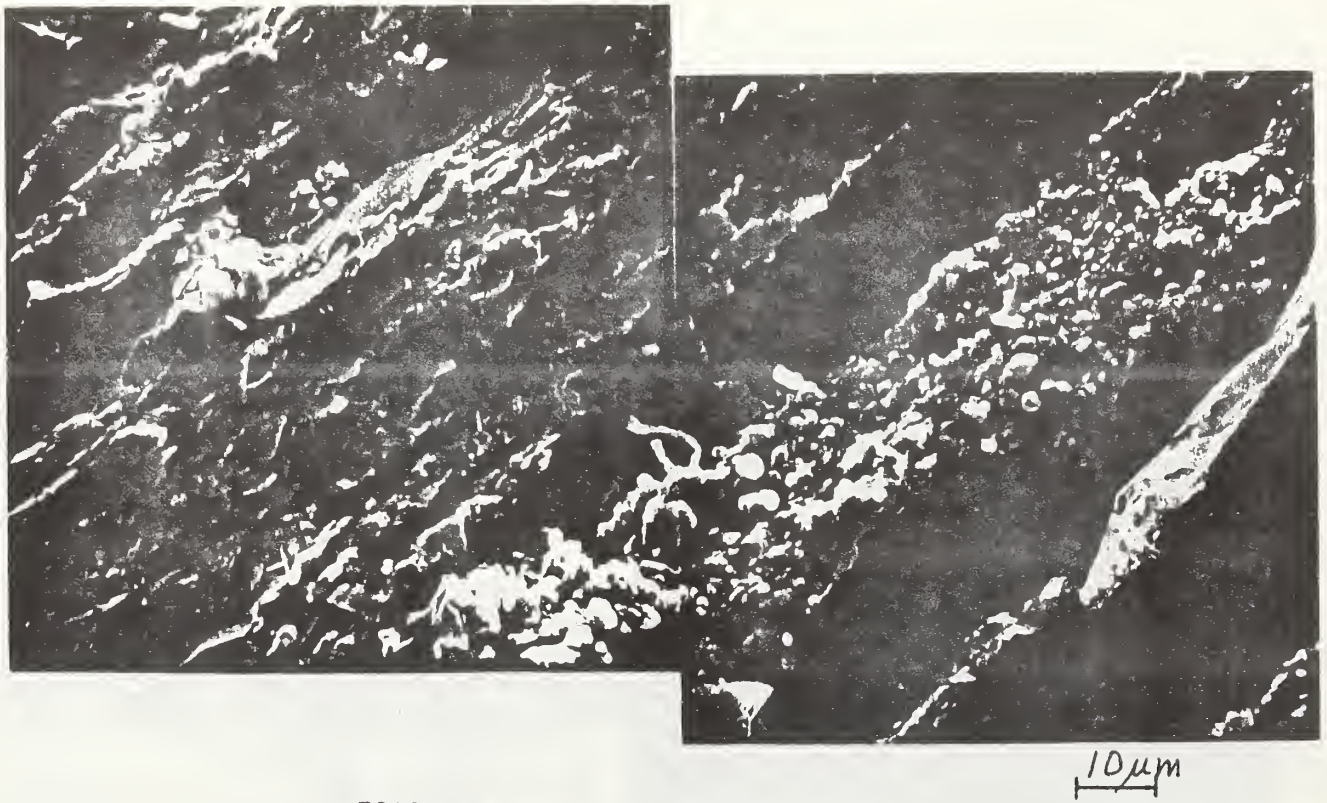


Fig. 8 F2925. Area of entrance deposit.

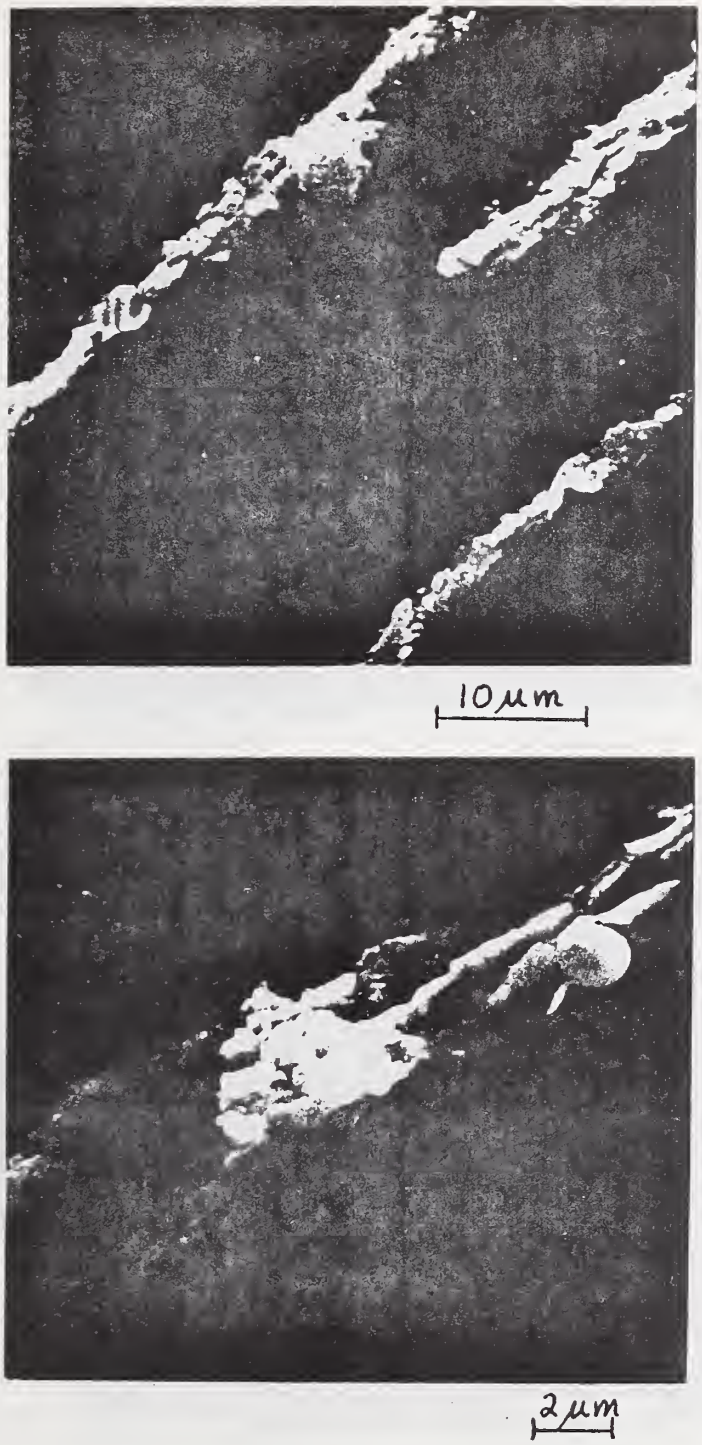


Fig. 9 F2925. Strings of wear particles. (a) ide particle (dark) is iron oxide with sulfur present. (b) Spheroid is Fe, as is end clump.

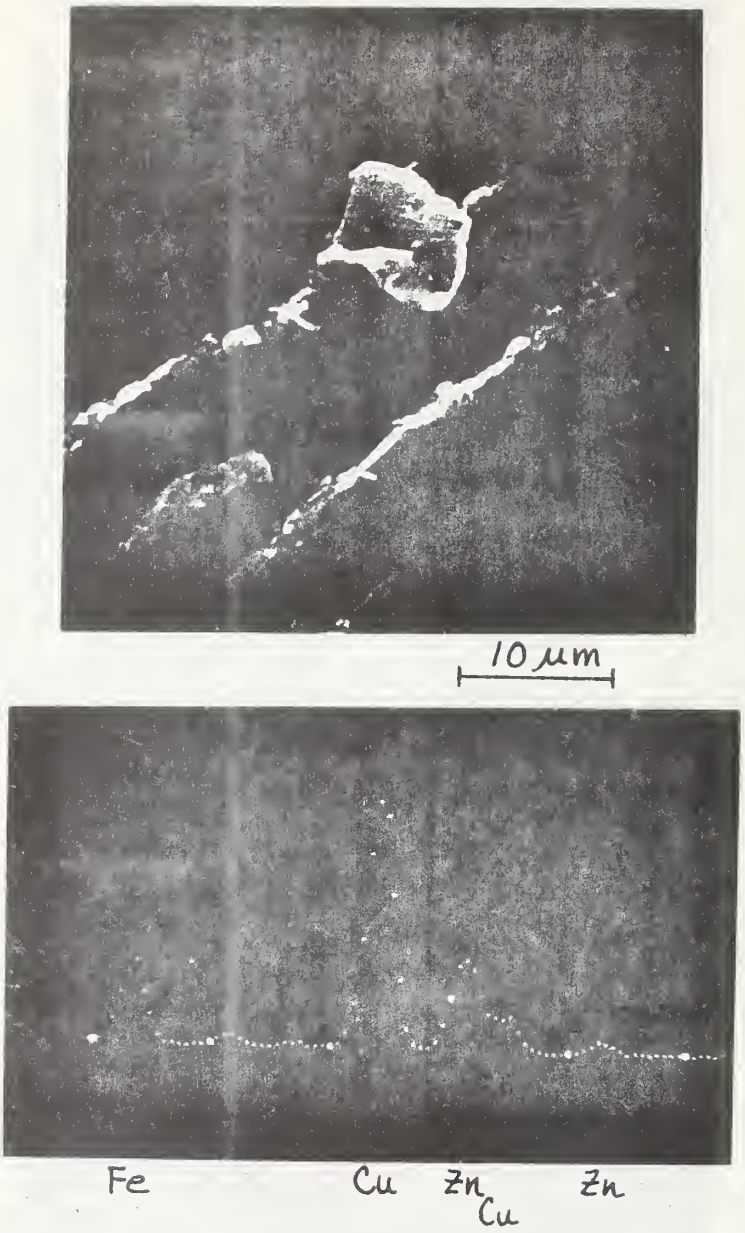


Fig. 10 F2925. Extraneous brass particle associated with iron particle string.

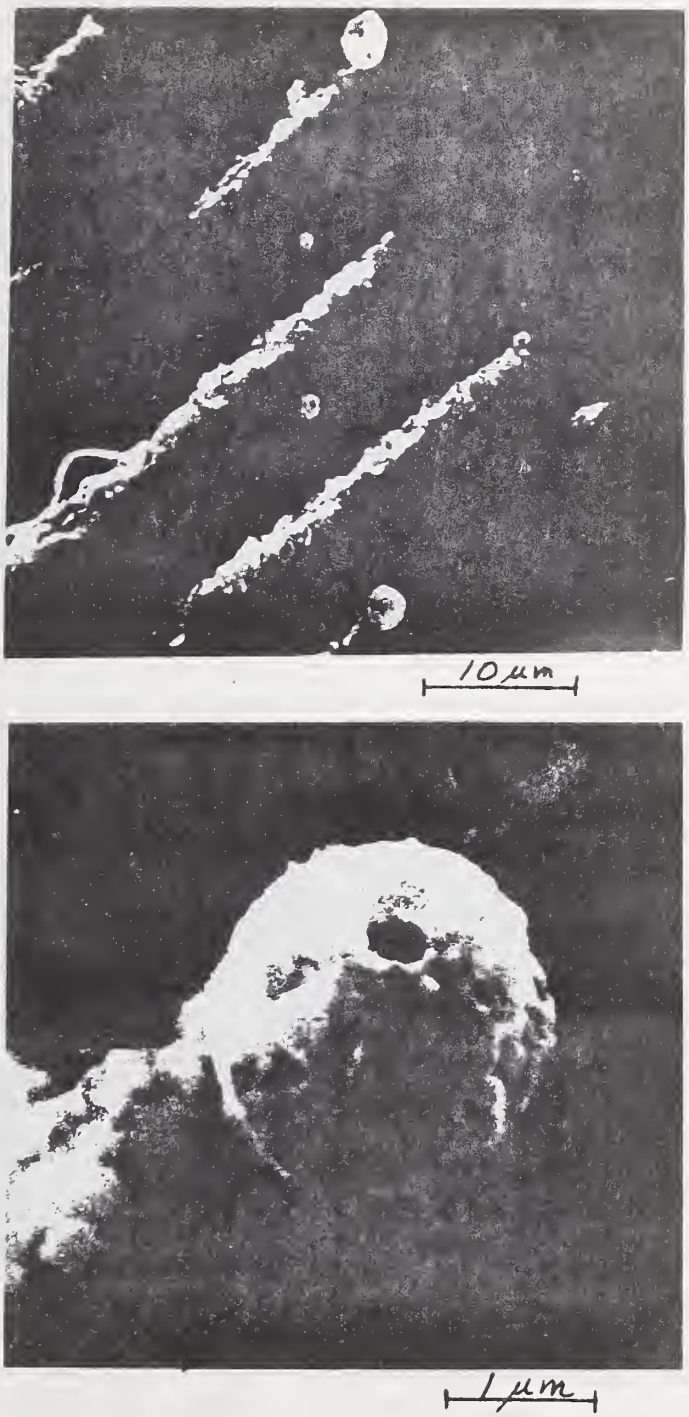
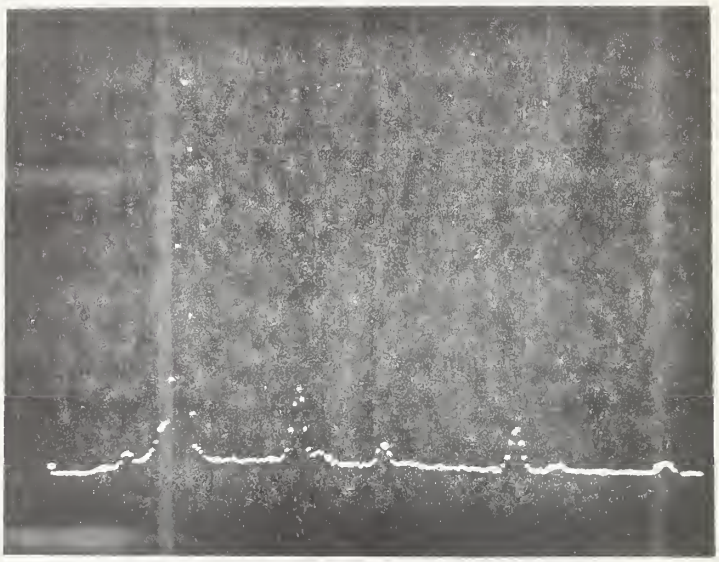
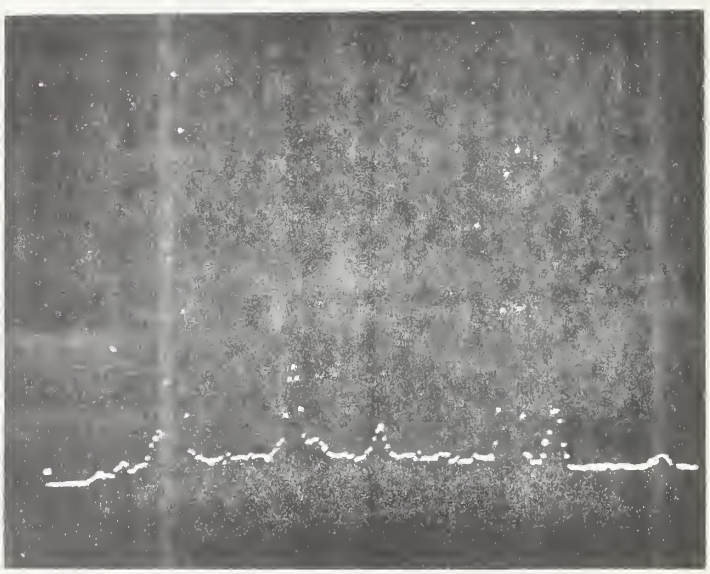


Fig. 11 F2916. Jet engine oil, bearing failure. Rough spheroidal particles.

Fig. 12 F2916. X-ray analysis of (a) spheroid and (b) iron wear particles. Full scale 5×10^3 cts.





	Si	K	Ti	Fe	Zn
Background	1.00	.33	.09	.06	.06
Spheroid (1)	1.00	.34	.11	.14	.04
Spheroid (7)	1.00	.18	.05	.12	.03
Iron particle	1.00	.24	.08	.82	.03

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15. SUPPLEMENTARY NOTES

16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

Results are presented from a program involved in characterizing the wear particles and surface degradation produced by wear in bearing and gear tests in which the effects of several variables on failure of the wearing surfaces has been examined. The information obtained has been correlated with the results of allied studies conducted by others in an attempt to develop an understanding of the processes producing wear and degradation of metal surfaces in sliding, rubbing, rolling, and/or rotating contact and the effects of lubricants, lubricant additives, bearing materials etc. on those processes. The characterization of the wear particles and wearing surfaces should aid in the establishment of the interrelationships between wear particle shape, size, size distribution, chemical compositions, metallurgical structure, and surface damage prior to failure.

17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)

Bearings; electron diffraction; electron microscopy; gears; lubrication; particles; wear

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